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STATE PATENT OFFICE
OF USSR (GOSPATENT)

SPECIFICATION OF INVENTION TO THE PATENT

METHOD OF A MANUFACTURE OF A SOFT CONTACT LENS

The essence of the invention: A method of a manufacture of a soft contact lens is in that acrylamide and N,N'–methylene–bis–acrylamide are preliminary subject to purification and then the starting solutions of acrylamide, N,N'–methylene–bis–acrylamide, N,N,N',N'–tetramethylethylenediamine and ammoniun persulfate are prepared at a concentration in a suitable solvent, in g/l:

acrylamide – 310.0 - 750.0;

N,N'–methylene–bis–acrylamide – 0.5 - 9.0;

N,N,N',N'–tetramethylethylenediamine – 0.1 - 3.0;

ammoniun persulfate – 0.1 - 4.0.

After preparing the starting solutions they are mixed and the polymerization is carried out in a closed volume having the shape of a contact lens (2 claims of the Claims; 1 Table).

DESCRIPTION

The invention relates to medicinal engineering, in particular, to ophthalmologic engineering, and may be used for the contact correction of vision.

The task of this invention consists in creating a method of manufacturing a soft contact lens in which the operations and the agents used in definite concentrations would improve the performance (service properties) of the soft contact lens due to increasing the physico-mechanical characteristics along with retaining high rate of moisture content and reducing allergic reactions upon application of said lens.

The set task is resolved by improving the method of a manufacture of a soft contact lens comprising the preparation of starting solutions of acrylamide, N,N'–methylene–bis–acrylamide, N,N,N',N'–tetramethylethylenediamine and ammoniun persulfate in a suit-able solvent, mixing thereof at a certain ratio and carrying out the polymerization in a closed

volume having the shape of a contact lens. According to the invention, the improvement consists in that acrylamide and N,N'–methylene–bis–acrylamide, before the preparation of the solutions, are subject to purification, and the solutions of acrylamide, N,N'–methylene–bis–acrylamide, N,N,N',N'–tetramethylethylenediamine and ammoniun persulfate are prepared at a concentration of, in g/l:

acrylamide	310.0 - 750.0;
N,N'–methylene–bis–acrylamide	0.5 - 9.0;
N,N,N',N'–tetramethylethylenediamine	0.1 - 3.0;
ammoniun persulfate	0.1 - 4.0.

The claimed method of manufacturing a soft contact lens secures the improvement of performance of a soft contact lens due to increasing the physico-mechanical characteristics along with retaining high rate of moisture content and reducing allergic reactions upon application of said lens.

The purpose is attained through proposed additional purification of acrylamide and N,N'- methylene-bis-acrylamide which permits to purify said monomers of acrylic acid residue, since removal of said acid from a gel structure is practically impossible, and presence of even traces of acrylic acid in a soft lens gives rise to allergic reaction of the eye mucous membrane. The operation of cleaning acrylamide and N,N'-methylene-bis-acrylamide of the acrylic acid traces is substantial indeed for the monomers prepared by various firms, since said undesirable elements are present therein. Moreover, acrylamide in the course of storage is partially polymerized, and presence of polyacrylamide in a monomer affects negatively the physico-mechanical characteristics of the resultant soft contact lens. N,N'-methylene-bis-acrylamide in the course of storage forms dimers and trimers, and presence of them in the initial solution also leads to deterioration of gel strength characteristics.

The increase of physico-mechanical characteristics is also provided by that the reagents solutions used in the method of the manufacture of the soft contact lens are prepared at the proposed concentrations.

It is feasible to carry out acrylamide and N,N'-methylene-bis-acrylamide purification by re-crystallization, hereupon, the most efficient purification rate is attained.

For the realization of the proposed method of the manufacture of the soft contact lens the following basic reagents are used: acrylamide, N,N'–methylene–bis–acrylamide, N,N,N',N'–tetramethylethylenediamine, ammoniun persulfate.

Acrylamide, C_3H_5NO, is used, having molecular weight of 71,08; white crystalline odorless powder; melting temperature 84,5±0,3^{0}C; density 1,122 g/cm^3; solubility in water at 25^{0}C – 215,5 g per 100 g of water; soluble in methanole, ethanole, acetone, chloroform, benzene. Basic substance content is 98,6%. Manufactured by "Reanal", Hungary, "Aldrich", USA.

N,N'-methylene-bis-acrylamide, $C_7H_9O_3$, molecular weight 154,16; white crystalline odorless powder; melting temperature 185^{0}C; solubility in water at the temperature of 20^{0}C – 3 g per 100 g of water. Basic substance content is 98%. Manufactured by "Reanal", Hungary, "Fluka Chemika"(Switzerland).

N,N,N',N'-tetramethylethylenediamine, $C_6H_{16}N_{12}$; molecular weight 116,21; colorless liquid, density 0,78 g/cm^3. Basic substance content 98,2%. Manufactured by "Reanal" (Hungary). Ammonium persulfate, molecular weight 228,19; colorless scaly crystals; density 1,982 g/cm^3; decomposition temperature 120^{0}C; solubility in water at 15,5^{0}C 74,8 g per 100 g of water. Basic substance content 98%. Manufactured by "Reanal", Hungary.

Before the preparation of the starting solutions additional purification of acrylamide and N,N'–methylene–bis–acrylamide is carried out. The purification is carried out, for example, by re-crystallization.

Acrylamide re-crystallization process is conducted as follows: 70 g of acrylamide is dissolved in 1 l of chloroform at 50-60°C, then the solution is filtered when hot. The filtrate is cooled in a freezer to (-15)-(-20)°C. The deposited crystals are filtered and washed on a filter with cool chloroform. After drying of the crystals melting temperature is determined. Basic substance content is 99%.

N,N'-methylene-bis-acrylamide re-crystallization process is conducted from acetone. For this purpose 30 g of N,N'-methylene-bis-acrylamide is dissolved in 1 l of acetone, refluxed, filtered through Shott filter, cooled to negative temperatures and crystals are filtered out. Melting temperature is determined. Basic substance content is 98%.

Then, the starting solutions of acrylamide, N,N'–methylene–bis–acrylamide, N,N,N',N'–tetramethylethylenediamine and ammoniun persulfate are prepared. For the preparation of the starting solutions a physiological solution or other suitable solvent, for example, distilled water, is used. The solution of acrylamide with the concentration of 310.0 - 750.0 g/l, the solution of N,N'–methylene–bis–acrylamide with the concentration of 0.5 - 9.0 g/l, the solution of N,N,N',N'–tetramethylethylenediamine with the

concentration of 0.1 - 3.0 g/l, the solution of ammoniun persulfate with the concentration of 0.1 - 4.0 g/l are prepared.

In the composition the ratio of N,N,N',N'–tetramethylethylenediamine to the mixture of acrylamide and N,N'–methylene–bis–acrylamide is used in such a way, that the volume ratio of N,N,N',N'–tetramethylethylenediamine to the volume of the mixture of acrylamide and N,N'–methylene–bis–acrylamide is from 1 : 6 to 1 : 25. The prepared composition for manufacturing the soft contact lens is placed into the form for carrying out the polymerization. The polymerization is carried out at a temperature of 20 – 25^{0}C for 45 to 60 minutes.

After completion of the polymerization process a soft contact lens is taken out of the mould, washed for 24 hours in a physiological solution, three-fold solution renewal. Upon this soft contact lens swelling to equilibrium state is completed.

With the soft contact lenses relative elongation, breaking strength, refraction coefficient, moisture content were determined. Breaking strength and relative elongation were determined on a modified instrument of Weiler-Rehbinder, the clamp separation velocity being 9,6 cm/min. Tests were conducted at the temperature of 20 ± 3^{0}C. Refraction index was determined with the help of refraction meter at the temperature of 20 ± 30^{0}C. Moisture content was determined by weight method by means of weighing equally swelled soft contact lenses and the lenses weighed to permanent weight. The lens power (in diopters) of soft contact lenses is determined with the help of lens meter.

EXAMPLE 1

The method of manufacturing a soft contact lens under the invention was implemented according to the technology disclosed above.

For manufacturing a soft contact lens acrylamide solution of 310 g/l concentration was used, along with solution of N,N'-methylene-bis-acrylamide with 9,0 g/l concentration, solution of N,N,N',N'-tetramethylethylenediamine with 0,1 g/l concentration, solution of ammonium persulfate with 4,0 g/l concentration.

The ratio of N,N,N',N'-tetramethylethylenediamine solution volume to the volume of the mixture of acrylamide and N,N'-ethylene-bis-acrylamide was 1:6. Time of polymerization was 60 minutes, polymerization temperature was 25^{0}C.

The resultant soft contact lens was of –3D.

In the resultant soft contact lens relative elongation was also determined, along with breaking strength, moisture content and refraction coefficient.

The results are given in Table 1.

EXAMPLE 2

The method of manufacturing a soft contact lens under the invention was implemented according to the technology disclosed above.

For manufacturing a soft contact lens the following solutions were used: solution of acrylamide with 750 g/l concentration, solution of N,N'-methylene-bis-acrylamide with 0,5 g/l concentration, solution of N,N,N'N'-tetramethyl-ethylenediamine with 3,0 g/l concentration, solution of ammonium persulfate with 0,1 g/l concentration.

The ratio of N,N,N',N'-tetramethyletylenediamine solution volume to the volume of the mixture of acrylamide and N,N'-methylene-bis-acrylamide was 1:11.

The time of polymerization was 45 minutes, temperature was 25^0C.

The resultant soft contact lens was of –10D.

In the resultant soft contact lens relative elongation was also determined, along with breaking strength, moisture content and refraction coefficient.

The results are given in the Table.

EXAMPLE 3

The method of manufacturing a soft contact lens under the invention was implemented according to the technology disclosed above.

For manufacturing a soft contact lens the following solutions were used: acrylamide solution of 520 g/l concentration, along with solution of N,N'-methylene-bis-acrylamide with 5,0 g/l concentration, solution of N,N,N',N'-tetramethylethylenediamine with 1,0 g/l concentration, solution of ammonium persulfate with 2,0 g/l concentration.

The ratio of N,N,N',N'-tetramethylethylenediamine solution volume to the volume of the mixture of initial solutions (acrylamide and methylene-bis-acrylamide was 1:7.

Time of polymerization was 50 minutes, polymerization temperature was 25^0C.

The resultant soft contact lens was of +6D.

In the resultant soft contact lens relative elongation was also determined, along with breaking strength, moisture content and refraction coefficient.

The results are given in Table.

EXAMPLE 4 (Comparative) The method of manufacturing a soft contact lens under the invention was implemented according to the technology disclosed above.

For manufacturing a soft contact lens acrylamide solution of 300 g/l concentration was used, along with solution of N,N'-methylene-bis-acrylamide with 9,0 g/l concentration, solution of N,N,N',N'-tetramethylethylenediamine with 0,1 g/l concentration, solution of ammonium persulfate with 4,0 g/l concentration.

The ratio of N,N,N',N'-tetramethylethylenediamine solution volume to the volume of the mixture of acrylamide and N,N'-ethylene-bis-acrylamide was 1:5.

Time of polymerization was 40 minutes, polymerization temperature was 25^0C.

The resultant soft contact lens was of +5D.

In the resultant soft contact lens relative elongation was also determined, along with break strength, moisture content and refraction coefficient.

The results are given in the Table.

EXAMPLE 5 (Comparative) The method of manufacturing a soft contact lens under the invention was implemented according to the technology disclosed above.

For manufacturing a soft contact lens the following solutions were used: acrylamide solution of 760 g/l concentration was used, along with solution of N,N'-methylene-bis-acrylamide with 10,0 g/l concentration, solution of N,N,N',N'-tetramethylethylenediamine with 4,0 g/l concentration, solution of ammonium persulfate with 5,0 g/l concentration.

The ratio of N,N,N',N'-tetramethylethylenediamine solution volume to the volume of the mixture of acrylamide and N,N'-ethylene-bis-acrylamide was 1:12.

Time of polymerization was 45 minutes, polymerization temperature was 25^0C.

The resultant soft contact lens was of –9,5D.

In the resultant soft contact lens relative elongation was also determined, along with breaking strength, moisture content and refraction coefficient.

The results are given in the Table.

EXAMPLE 6 (Comparative) The method of manufacturing a soft contact lens under the invention was implemented according to the technology disclosed above.

For manufacturing a soft contact lens acrylamide solution of 500 g/l concentration was used, along with solution of N,N'-methylene-bis-acrylamide with 0,4 g/l concentration, solution of N,N,N',N'-tetramethylethylenediamine with 0,05 g/l concentration, solution of ammonium persulfate with 0,06 g/l concentration.

The ratio of N,N,N',N'-tetramethylethylenediamine solution volume to the volume of the mixture of acrylamide and N,N'-ethylene-bis-acrylamide was 1:7.

Time of polymerization was 45 minutes, polymerization temperature was 25^0C.

The resultant soft contact lens was of 0,0D.

In the resultant soft contact lens relative elongation was also determined, along with breaking strength, moisture content and refraction coefficient.

The results are given in the Table.

It is evident from Table 1 the soft contact lenses manufactured in accordance with the proposed method have higher physico-mechanical characteristics with high moisture content in comparison with the soft contact lenses manufactured in compliance with SU A 959313.

Comparative Examples (Examples 4-6) indicate that quite substantial in manufacturing of the soft contact lenses under the claimed method is the use of the initial solutions with the claimed concentrations, as well as definite ratios in the reaction mixture thereof, since alteration of said concentrations and ratios leads to reduction of physico-mechanical and optic characteristics (Example 4), or deterioration of surface quality in the soft contact lens (Example 5).

Consequently, the above mentioned Examples are eventually some of concrete Examples of embodiment of invention. However, it is evident that other modifications which do not substantially alter the invention are also possible.

CLAIMS

1. A method of a manufacture of a soft contact lens which comprises that the starting solutions of acrylamide, N,N'–methylene–bis–acrylamide, N,N,N',N'–tetra-methylethylenediamine and ammoniun persulfate are prepared in a suitable solvent, they are mixed at a certain ratios and polymerization in a closed volume having the shape of a contact lens is carried out, said method ***characterized*** in that acrylamide and N,N'–methylene–bis–acrylamide, before the preparation of the solutions, are preliminary subject to purification; and the solutions of acrylamide, N,N'–methylene–bis–acrylamide, N,N',N'–tetra-methylethylenediamine and ammoniun persulfate are prepared at a concentration of, in g/l: acrylamide – 310.0 - 750.0; N,N'–methylene–bis–acrylamide – 0.5 - 9.0; N,N,N',N'–tetramethylethylenediamine – 0.1 - 3.0; ammoniun persulfate – 0.1 - 4.0.

2. A method according to claim 1, ***characterized*** in that a purification of acrylamide and N,N'–methylene–bis–acrylamide is carried out by recrystallization.

Table 1

Index	According to invention Examples			Comparative Examples Examples			According to A 959313
	1	2	3	4	5	6	
Relative elongation,%	210,0	320,0	300,0	240,0	Surface quality is not satisfactory	370,0	150,0
Breaking strength,kPa	173,0	154,0	97,0	129,0		93,0	90,0
Moisture content, %	87,0	81,0	90,0	88,0		90,0	90,0
Refraction Coefficient	1,355	1,370	1,353	1,355		1.343	1,336